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Environmental Cleanup Program

Environmental Restoration Project
Standard Operating Procedure

for:

Routine Validation of Chemical Separation Alpha Spectrometry, Gas Proportional Counting, and Liquid Scintillation Data

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Routine Validation of Chemical Separation Alpha Spectrometry, Gas Proportional Counting, and Liquid Scintillation Data

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List of Acronyms and Abbreviations

CLP	Contract Laboratory Program (EPA)
COC	chain of custody
DER	duplicate error ratio
EPA	US Environmental Protection Agency
ER	environmental restoration
FSF	Field Support Facility
GPC	
LANL	Los Alamos National Laboratory
LCS	laboratory control sample
MDC	minimum detectable concentration
n/a	not applicable
N.A.	not available
%R	percent recovery
QC	quality control
RER	replicate error ratio
RN	request number
σ	sigma (standard deviation of a set of measurements)
SMO	Sample Management Office
SOP	standard operating procedure
SOW	statement of work
TPU	total propagated uncertainty

Routine Validation of Chemical Separation Alpha Spectrometry, Gas Proportional Counting, and Liquid Scintillation Data

NOTE: Environmental Restoration (ER) Project personnel may produce paper copies of this procedure printed from the controlled-document electronic file located at http://erinternal.lanl.gov/documents/Procedures/sops.htm. However, it is their responsibility to ensure that they are trained to and utilizing the current version of this procedure. The author may be contacted if text is unclear.

1.0 PURPOSE

This standard operating procedure (SOP) represents the minimum standard for evaluating routine radionuclide analytical data generated for the Los Alamos National Laboratory (LANL) ER Project for samples analyzed for

- alpha-emitting isotopes (americium-241; uranium-234, -235, and -238; and plutonium-238 and -239/240) by chemical separation alpha spectrometry,
- strontium-90 by gas proportional counting (GPC),
- gross alpha and beta analyses by GPC,
- tritium by liquid scintillation, and
- gross gamma-emitting isotopes by sodium iodide detector

using the methods required under the current statement of work (SOW) for analytical services (LANL 1995). Because each method has different requirements due to the different technologies used, the validation procedure in Section 6.0 provides direction for applying the requirements in each subsection to the different methods and isotopes. The evaluation of data by this procedure is not specific to a particular data use, although this procedure may be used as a point of departure for developing focused data-validation requirements specific to a particular data use.

Note: Implementation of this procedure results in a tabulation of data compliances and noncompliances identified relative to expectations based on national guidelines (EPA 1994) for data review. Because the US Environmental Protection Agency (EPA) guidelines are specific to analyses for inorganic chemicals, additional guidance (ANSI 1996, Currie 1968, Fong and Alvarez 1996, MARSSIM 1997, and LANL 2000) was employed in the preparation of this SOP. Because the acceptance criteria used for this procedure are not based on site-specific acceptance criteria, the results of this validation

procedure are intended to be used as *general indicators* of data quality and should not be construed as a definitive identification of data usability.

Note: Implementation of this procedure may be followed by a more focused and data use-specific evaluation of data, especially if implementation of this SOP indicates that technical deficiencies may exist in the data.

2.0 TRAINING

All data validators who implement this SOP shall possess a minimum of a Bachelors degree in chemistry and two years experience in generating analytical data in an environmental analytical laboratory, or two years of data validation experience. New validators shall work under the direct supervision of an experienced ER Project validator. The work of new validators shall be reviewed and signed by an experienced ER Project validator until ten data packages for each analytical suite have been satisfactorily validated. ER Project validators shall have demonstrated familiarity with the EPA national functional guidelines for data review. All data validators must document that they have read and understand this SOP and completed all applicable training assignments in accordance with QP-2.2.

3.0 DEFINITIONS

3.1 *Activity concentration* — Level of radioactivity per unit volume or mass measured as a concentration; usually reported in pCi/g or pCi/L.

3.2 *Analyte* — The element, nuclide, or ion a chemical analysis seeks to identify and/or quantify; the chemical constituent of interest.

3.3 *a posteriori* — In this SOP, defined as “after the measurement.”

3.4 *a priori* — In this SOP, defined as “before the measurement.”

3.5 *Blank sample* — Sample expected to have negligible or unmeasurable amounts of analytes. Results of blank sample analyses indicate whether field samples might have been contaminated during the sample collection, transport, storage, preparation, and analysis process.

3.6 *Data validator* — A person who has met the minimum standards of training established by the ER Project for data validation and who performs data validation on behalf of the ER Project.

3.7 *Decision level concentration* — Activity concentration level used to classify radiochemical measurements as “detected” or “nondetected” (e.g., measured results above the DLC are “detected”). The DLC is established from an appropriate blank count at the 0.05-significance level. Therefore an observed activity concentration above the DLC has a less than 5% chance of originating from a sample containing no (or blank) levels of analyte.

$$DLC = C \times 2.33\sqrt{N_b} = C \times 2.33 S_b .$$

Where:

DLC = decision level concentration reported in pCi/g or pCi/L,

C = a group of factors that convert counts to an activity concentration (C is omitted if S_b is expressed in concentration units),

2.33 = 1.65 $(2)^{0.5}$ (1.65 normal probability, one sided, for 0.05 significance),

N_b = total analyte-free blank (or background) count,

S_b = standard deviation of the blank count, and

all blank, background, and sample count times are equal.

3.8 *Detect (radionuclides)* — Sample result greater than the minimum detectable concentration (MDC) reported by the analytical laboratory. The laboratory reports the concentration of the analyte in the sample.

3.9 *Detector background* — Ambient signal response, recorded by radioactivity measuring instruments, that is independent of radioactivity contributed by the radionuclides being measured in the sample.

3.10 *Duplicate analysis* — Analysis performed on one of a pair of identically prepared subsamples taken from the same sample.

3.11 *Duplicate error ratio (DER)* — See replicate error ratio (RER).

3.12 *Holding time* — The maximum elapse of time that one can expect to store a sample without unacceptable changes in analyte concentrations. Holding times apply under prescribed conditions and deviations from these conditions may affect the holding time. Extraction holding time refers to the time lapse from sample collection to sample preparation; analytical holding time refers to the time lapse between sample preparation and analysis.

3.13 *Laboratory control sample (LCS)* — A known matrix that has been spiked with compound(s) representative of the target analytes. The LCS is used to document laboratory performance. The acceptance criteria for LCSs are method specific.

3.14 *Laboratory duplicate sample* — The portions of a sample taken from the same sample container, prepared for analysis and analyzed independently but under identical conditions; used to assess or demonstrate acceptable laboratory method precision at the time of analysis. Each duplicate sample is expected to be equally representative of the original material. Duplicate analyses also are performed to generate data, to determine the long-term precision of an analytical method on various matrices.

3.15 *LANL data validation qualifiers* — The data qualifiers defined by the Laboratory (LANL) and used in the ER Project baseline-validation process.

For a complete list of data qualifiers applicable to any particular analytical suite, consult the appropriate ER Project SOP.

3.16 *LANL data validation reason codes* — The codes applied to the sample data by data validators who are independent of the contract laboratory which performed the sample analysis. Reason codes provide an in-depth and analysis-specific explanation for applying the qualifier with some description of the potential impact on the data use. For a complete list of data qualifiers applicable to any particular analytical suite, consult the appropriate ER Project SOP.

3.17 *Matrix spike* — An aliquot of sample spiked with a known concentration of target analyte(s). Matrix spike samples are used to measure the ability to recover prescribed analytes from a native sample matrix. The spiking typically occurs before sample preparation and analysis.

3.18 *Minimum detectable concentration* — Minimum activity concentration that the analytical laboratory equipment can detect in 95% of the analyzed samples. That is, if the actual concentration of a sample is above MDC, a less than a 5% chance exists that the measured concentration will fall below the DLC and result in a "nondetect." An MDC measures analytical performance (not detection limits).

$$MDC = C \times (2.71 + 4.65\sqrt{N_b}) .$$

Where:

MDC = minimum detectable concentration reported in pCi/g or pCi/L,

C = a group of factors that convert counts to an activity concentration (C is omitted if S_b is expressed in concentration units),

2.71 = 1.65^2 (1.65 normal probability, one sided, for 0.05 significance),

4.65 = 1.65 x $2(2)^{0.5;}$

N_b = total analyte-free blank (or background) counts, and

all blank, background, and sample count times are equal.

3.19 *Non-detect* – A sample result that is less than the MDC.

3.20 *Percent recovery (%R)* — Amount of material detected in a sample (minus any amount already in the sample) divided by the amount added to the sample and expressed as a percentage.

3.21 *Preparation blank* — An analyte-free matrix to which all reagents are added in the same volumes or proportions as those used in the environmental sample processing, and which is prepared and analyzed in the same manner as the corresponding environmental samples. The preparation blank is used to assess the potential for contamination of samples during preparation and analysis.

3.22 *Replicate error ratio* — Measure of precision of analytical laboratory replicate samples in a batch. The RER is based on the standard deviations of the sample and the replicate sample.

$$\text{RER} = \frac{S - R}{\sqrt{u_S^2 + u_R^2}}.$$

Where:

RER = replicate error ratio,
S = sample value,
R = replicate value,
u_S = sample uncertainty, and
u_R = replicate uncertainty.

If |RER| < 2, then the sample and replicate are not statistically different at the 95% confidence level.

3.23 *Request number (RN)* — An identifying number assigned by the ER Project to a group of samples that are submitted for analysis.

3.24 *Routine data* — Data generated using analytical methods that are identified as routine methods in the current ER Project SOW for analytical services.

3.25 *Routine data validation* — The process of reviewing analytical data relative to quantitative routine acceptance criteria. The objective of routine data validation is two-fold: one objective is to estimate the technical quality of the data relative to minimum national guidelines adopted by the ER Project; the other objective is to indicate to data users the technical data quality at a general level by assigning qualifier flags to environmental data whose quality indicators do not meet acceptance criteria.

3.26 *Routine radionuclide data* — Analytical results and associated data for samples analyzed for alpha-emitting isotopes (by chemical-separation alpha spectrometry), strontium-90 (gas proportional counting or GPC), gross alpha and beta analyses (gas proportional counting), and tritium (by liquid scintillation). Routine validation of gamma spectroscopy is included in ER-SOP-15.06 and not in ER-SOP-15.07 because of the greater complexity of gamma spectroscopy data.

3.27 *Sigma (s)* — Standard deviation (square root of the variance) of a set of measurements. For normally distributed data, a range of one sigma (1σ) below the estimated mean to one sigma (1σ) above the estimated mean signifies a 67% confidence that the mean of a population lies within that range. Similarly, a range of plus/minus 2 sigma (±2σ) implies 95% confidence that a population mean lies within that range.

3.28 *Target analyte* — An element, chemical, or parameter, the concentration, mass, or magnitude of which is designed to be quantified by use of a particular test method.

3.29 *Total propagated uncertainty (TPU)* — Sum of all aspects of uncertainty introduced throughout the sample analysis process, from sample collection to reporting of results. Many aspects of TPU may be specifically calculated by an analytical laboratory (e.g., net instrumental error, counting uncertainty). Other aspects of TPU may not be quantifiable (e.g., heterogeneity of concentrations at site), and thus cannot be directly included in a laboratory's estimate of TPU.

4.0 BACKGROUND AND PRECAUTIONS

4.1 To protect the integrity of the data record package, the **data validator** must store and handle all data record packages under ER Project chain-of-custody (COC) rules in accordance with ER-SOP-15.09.

4.2 Logic diagrams that appear in this SOP are included for experienced validators to expedite the validation process and do not include instructions for where to record validation results. Those instructions may be found in the SOP text that corresponds to each logic diagram.

4.3 The chemical separation alpha spectrometry, gas proportional counting, and liquid scintillation data validation checklist forms (hereafter referred to as the routine radionuclide data validation checklist) require actions to be taken if a particular validation condition is true or false. It is important to look at the top of each validation form to know whether action is required when the condition is true or when the condition is false.

4.4 The validation process requires that the **data validator** record LANL data validation qualifiers and reason codes on photocopies of the data summary results forms ("Form Is") in the hard copy data record packages. Contiguous lines of identical qualification on the photocopied Form Is may be represented as the qualifier flag and reason code, followed by a vertical downward arrow to the end of the block of results that are qualified identically.

4.5 The routine radionuclide data validation checklists of Attachment D are examples of the actual forms to be used for data validation under this SOP. Individual parts of the forms may be reproduced as necessary to complete the validation of a data record package.

5.0 EQUIPMENT

The **validator** may need the following equipment and supplies to implement this procedure:

5.1 current routine radionuclide data validation checklist forms (see examples in Attachment D),

5.2 data record packages to be validated,

5.3 electronic calculator (optional),

5.4 photocopier, and

5.5 current ER Project SOW for analytical services.

6.0 PROCEDURE

Note: Deviations from this SOP are made in accordance with QP-4.2.

Note: While this SOP is applicable to chemical-separation alpha spectrometry (for alpha-emitting isotopes) GPC (for strontium-90 and gross α/β), and liquid scintillation (for tritium), each subsection does not apply equally to each method. Notes at the beginning of each subsection describe the applicability of each subsection to each method.

6.1 Prepare for Data Validation:

Note: Section 6.1 applies to all routine radionuclide analysis methods and analytes. One copy of the validation forms will be needed for each routine radionuclide method reported.

6.1.1 The **validator** will begin by obtaining the required current versions of the routine radionuclide data validation checklist forms (see Attachment D) from the ER Project website (http://erinternal.lanl.gov/Quality/forms.htm).

6.1.2 Obtain from the Sample Management Office (SMO) of the Field Support Facility (FSF) the data record package(s) that contain the sample data to be validated.

6.1.3 Prepare a data validation cover sheet (see Attachment C) by filling out the top part of the form and placing a check or other mark adjacent to the analytical suite(s) for which this validation is being performed.

Note: A single cover sheet may be used for validation of multiple analytical suites under the same RN.

Note: Use a separate sheet of paper to document each deficiency identified beyond the scope of this procedure including phone conversations

with the analytical laboratory personnel concerning these deficiencies. Attach these sheets to the data validation cover sheet.

6.1.4 Verify that the following items are present in the data record package:

6.1.4.1 signed LANL COC record;

6.1.4.2 case narrative;

6.1.4.3 result forms (Contract Laboratory Program [CLP] Form I or equivalent) for each sample;

6.1.4.4 quality control (QC) forms (CLP forms, or equivalent) for water and/or soils, as appropriate.

6.1.4.5 instrument readout (raw data) for the samples.

6.1.5 If the data record package does not contain all items listed in Sections 6.1.4.1 through 6.1.4.5, contact the analytical laboratory to obtain those materials.

6.1.5.1 If required documentation is missing from the data record package, and the package is less than six months old, contact the analytical laboratory and allow three business days for the laboratory to submit the required documentation.

6.1.5.2 If the analytical laboratory does not submit documentation within three business days, return the data record package to the SMO for contract-compliance action.

6.1.5.3 If the data record package is greater than 6 months old, allow 10 business days for the analytical laboratory to submit the required documentation before returning the data record package to the SMO.

6.1.6 Record the presence or absence (Y or N) of each item, as appropriate, in the completeness checklist of the data validation cover sheet.

6.1.7 In the data validation cover sheet completeness checklist section, note any samples whose data are missing from the data record package.

6.1.8 Photocopy all analytical laboratory QC forms from the data record package.

6.1.9 Photocopy the case narrative from the data record package.

6.1.10 Photocopy the Form Is to be used during the validation process before you begin completing this procedure.

Caution: Do not record data-validation qualifiers and reason codes on the original form (Form Is).

Note: The **validator** must submit photocopies of the items listed in Sections 6.1.8 through 6.1.10 as attachments to the completed data validation checklists.

6.2 Verify Sample Detect Status and Statistically Validate Sample Results

Note: Section 6.2 applies to all routine radionuclide analysis methods and analytes. This analysis must be performed first in order to determine the detection status for the target analytes before subsequent steps in this validation procedure.

$$MDC = C \times (2.71 + 4.65\sqrt{N_b}) . \qquad \text{(Equation 1)}$$

6.2.1 If a MDC *was stated* in the report for each nuclide in each batch associated with this RN,

6.2.1.1 record “Y” in block 1a of the routine radionuclide data validation checklist, Part I;

6.2.1.2 record the applicable nuclides and MDCs in block 1c of the routine radionuclide data validation checklist, Part I;

6.2.1.3 record “n/a” in blocks 2a and 2c of the routine radionuclide data validation checklist, Part I; and

6.2.1.4 go to Section 6.2.3.

6.2.2 If a MDC *was not stated* in the report for each nuclide in each batch associated with this RN,

6.2.2.1 record “N” in block 1a of the routine radionuclide data validation checklist, Part I and

6.2.2.2 record “N.A.” in block 1c of the routine radionuclide data validation checklist, Part I.

6.2.2.3 record “Y” in block 2a of the routine radionuclide data validation checklist, Part I;

6.2.2.4 calculate an estimated sample-specific MDC as 3σ of the sample result; and

6.2.2.5 record this value appropriately in block 2c of the routine radionuclide data validation checklist, Part I.

6.2.3 If the sample value is less than the MDC,

6.2.3.1 record “Y” in block 3a of the routine radionuclide data validation checklist, Part I;

6.2.3.2 circle “U, R5” in block 3b of the routine radionuclide data validation checklist, Part I;

6.2.3.3 record the qualifier flag and reason code combination “U, R5” next to the result for each affected target analyte, on Form I; and

6.2.3.4 record what analytes were qualified in block 3c on the routine radionuclide data validation checklist, Part I.

6.2.4 If the sample value is greater than the MDC,

6.2.4.1 record “N” in block 3a of the routine radionuclide data validation checklist, Part I and

6.2.4.2 record “n/a” in block 3c of the routine radionuclide data validation checklist, Part I.

6.2.5 Find the reported DER in the data record package for the duplicate and the sample result, or calculate the DER (or RER) as follows:

$$\text{RER} = \frac{S - R}{\sqrt{u_S^2 + u_R^2}}. \qquad \text{(Equation 2)}$$

Where:

RER = replicate error ratio,

S = sample value,

R = replicate value,

u_S = sample uncertainty, and

u_R = replicate uncertainty.

If |RER| is less than 2, then the sample and replicate are not statistically different at the 95% confidence level.

6.2.6 If the DER is less than or equal to 2 for the sample nuclide and the sample result,

6.2.6.1 record “N” in block 4a of the routine radionuclide data validation checklist, Part I;

6.2.6.2 “n/a” in block 4c of the routine radionuclide data validation checklist, Part I; and

6.2.6.3 go to Section 6.3, Verify Method-Blank Results

6.2.7 If the DER is greater than 2 for the sample nuclide and the sample result,

6.2.7.1 record “Y” in block 4a of the routine radionuclide data validation checklist, Part I;

6.2.7.2 circle “R, R7b” in block 4b of the routine radionuclide data validation checklist, Part I;

6.2.7.3 record the qualifier flag and reason code combination "R, R7b" next to the result for each affected target analyte, on Form I; and

6.2.7.4 record what analytes were qualified in block 4c of the routine radionuclide data validation checklist, Part I.

6.2.8 Use the logic diagram in Figure 6.2-1 to determine which, if any, LANL qualifier flags and reason codes the **validator** must assign to the sample results based on the sample detect status validation of the sample results and on the statistical analysis of the sample results.

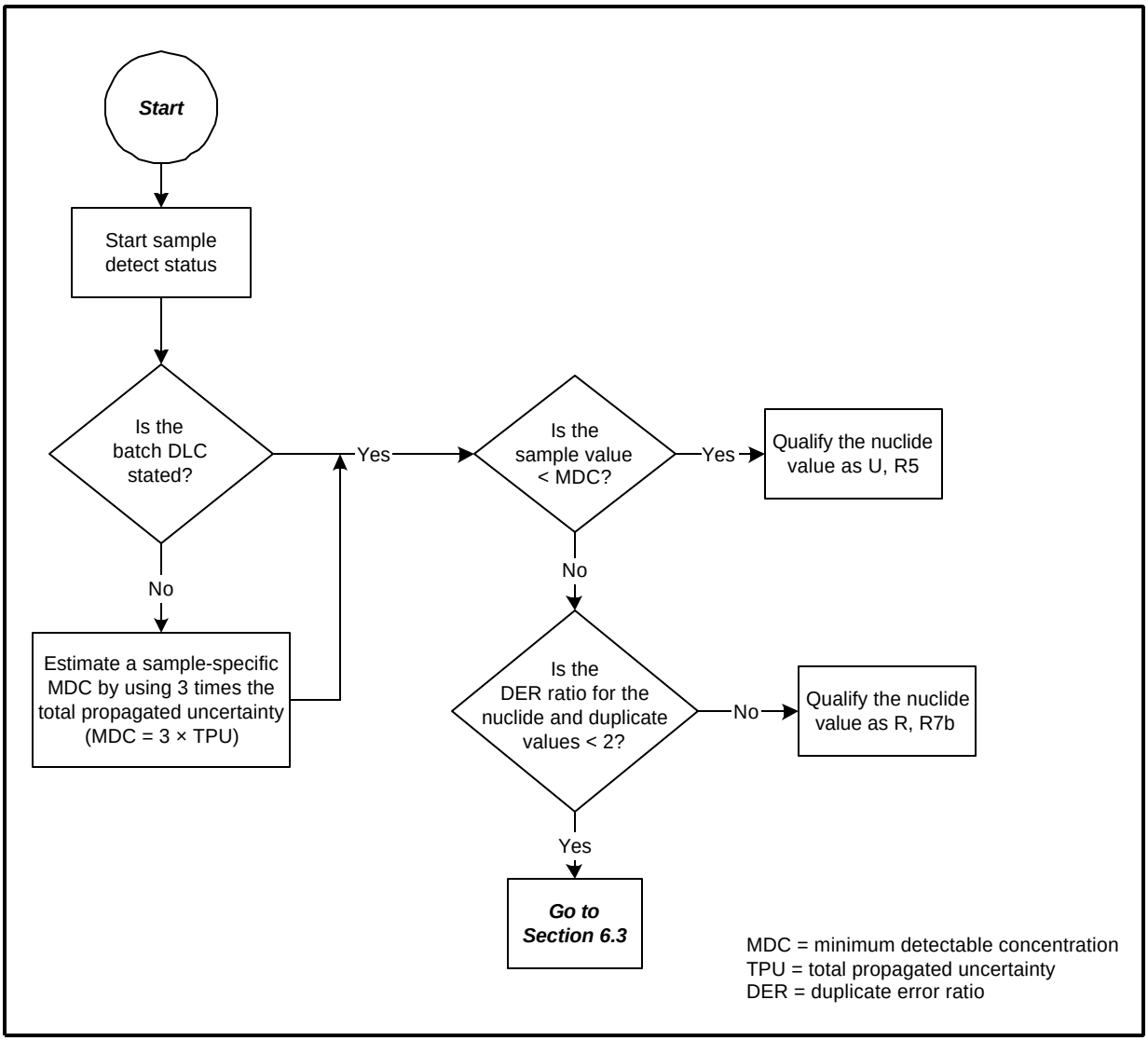

Figure 6.2-1. Assigning LANL qualifier flags and reason codes to the sample results based on the sample detect status validation of the sample results and on the statistical analysis of the sample results.

6.3 Verify Method-Blank Results

Verify the presence of the required blanks and their associated results using forms provided by the analytical laboratory.

Note: Section 6.3 applies to all routine radionuclide analysis methods and analytes. The method-blank results must be less than the MDC for each nuclide.

Note: If additional validation forms are needed to record validation data for more than one blank, make additional copies of the appropriate forms.

6.3.1 If a method blank *was analyzed* for each sample matrix and/or each specified nuclide in each batch associated with this RN,

6.3.1.1 record “Y” in block 1a of the routine radionuclide data validation checklist, Part IIa;

6.3.1.2 record “n/a” in block 1c of the routine radionuclide data validation checklist, Part IIa; and

6.3.1.3 go to Section 6.3.3.

6.3.2 If a method blank *was not analyzed* for each sample matrix and/or each specified nuclide in each batch associated with this RN,

6.3.2.1 record “N” in block 1a of the routine radionuclide data validation checklist, Part IIa;

6.3.2.2 circle “A, R4b” in block 1b of the routine radionuclide data validation checklist, Part IIa;

6.3.2.3 record the qualifier flag and reason code combination “A, R4b” next to each affected sample result, on Form I; and

6.3.2.4 record the affected samples in block 1c of the routine radionuclide data validation checklist, Part IIa. No further qualification is done.

6.3.3 If the method-blank result is greater than the MDC for *any* analytes,

6.3.3.1 go to Section 6.3.5.

6.3.4 If the method-blank result is less than the MDC for *any* analyte(s),

6.3.4.1 record “N” in block 2a of the routine radionuclide data validation checklist, Part IIb;

6.3.4.2 record “n/a” in blocks 2c and 2d of the routine radionuclide data validation checklist, Part IIb; and

6.3.4.3 go to Section 6.4, Verify Tracer/Carrier Recovery.

6.3.5 For those analytes that are greater than the MDC in the blank, if the analytes detected in the sample are greater than 5 times the blank level,

6.3.5.1 record “N” in block 2a of the routine radionuclide data validation checklist, Part IIb and

6.3.5.2 go to Section 6.4, Verify Tracer/Carrier Recovery.

6.3.6 For those analytes that are greater than the MDC in the blank, if the analytes detected in the sample at less than 5 times the blank level,

6.3.6.1 record “Y” in block 2a of the routine radionuclide data validation checklist, Part IIb;

6.3.6.2 circle U, R4 in block 2b of the routine radionuclide data validation checklist, Part IIb;

6.3.6.3 record the qualifier flag and reason code combination “U, R4” next to the result for each affected target analyte, On Form 1; and

6.3.6.4 record what analytes were qualified in block 2c and the analyte concentrations in block 2d of the routine radionuclide data validation checklist, Part IIb.

6.3.7 Use the logic diagram of Figure 6.3-1 determine which, if any, LANL qualifier flags and reason codes the **validator** must assign to the sample results for noncompliant blanks.

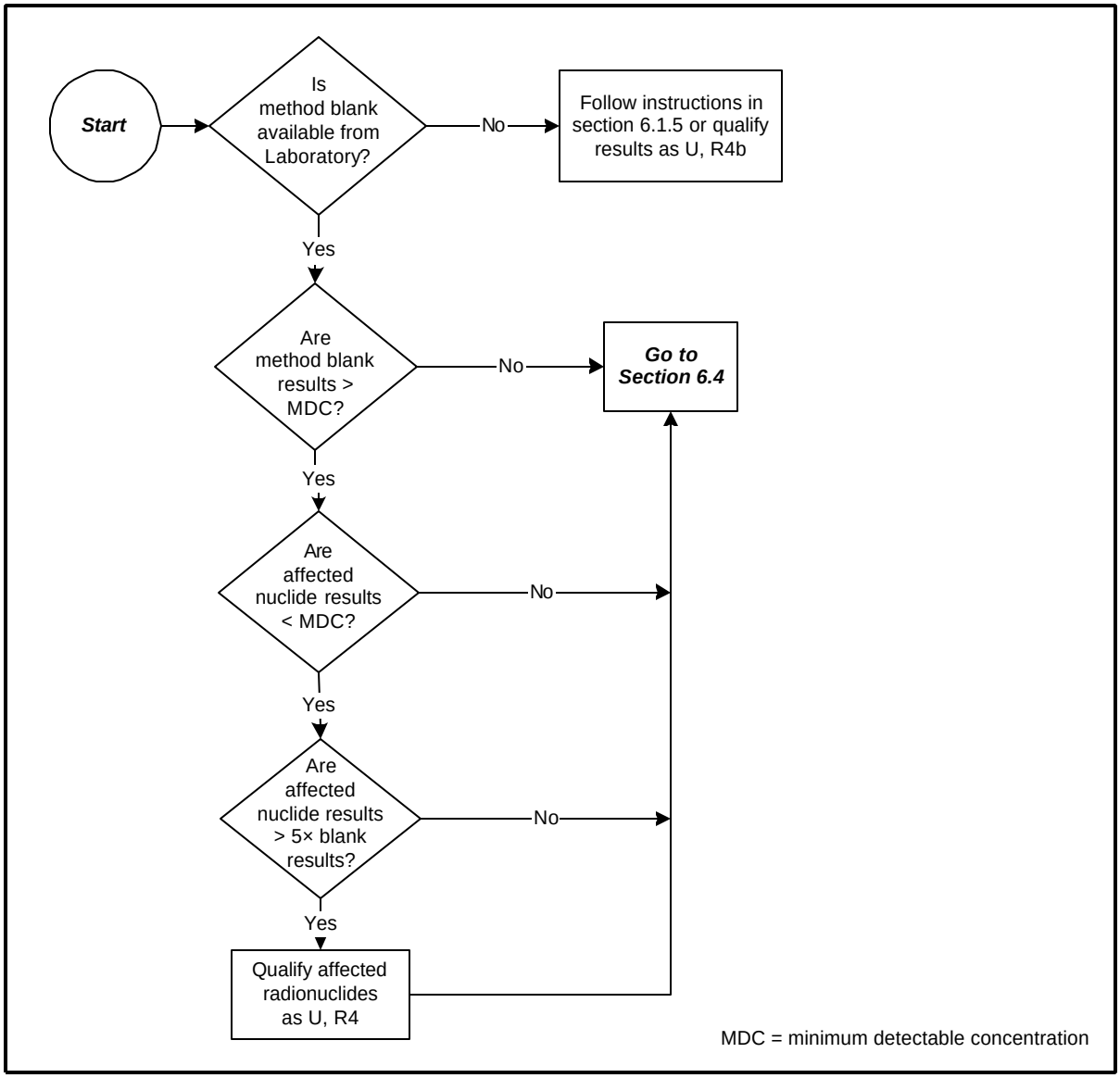

Figure 6.3-1. Assigning LANL qualifier flags and reason codes to the sample results for noncompliant blanks.

6.4 Verify Tracer/Carrier Recovery

Note: Section 6.5 only applies to chemical separation alpha spectrometry and GPC for Strontium-90. This section does not apply to tritium by liquid scintillation or to gross α/β by GPC. For tritium and gross α/β, go to Section 6.5.

6.4.1 If the tracer/carrier %R value *is reported* for each specified nuclide,

6.4.1.1 record a "Y" in block 1a of the routine radionuclide data validation checklist, Part IIIa;

6.4.1.2 record "n/a" in block 1a of the routine radionuclide data validation checklist, Part IIIa; and

6.4.1.3 go to Section 6.4.3.

6.4.2 If the tracer/carrier %R value *is not reported* for each specified nuclide,

6.4.2.1 record “N” in block 1a of the routine radionuclide data validation checklist, Part IIIa.

6.4.2.2 circle A, R1e in block 1b of the routine radionuclide data validation checklist, Part IIIa;

6.4.2.3 record the qualifier flag and reason code combination “A, R1e” next to the result for each affected target analyte, on Form I; and

6.4.2.4 record what samples were qualified in block 1c of the routine radionuclide data validation checklist, Part IIIa.

6.4.3 If the tracer/carrier %R value is greater than 30%,

6.4.3.1 record “N” in blocks 2a and 3a of the routine radionuclide data validation checklist, Part IIIb;

6.4.3.2 record “n/a” in blocks 2c and 3c of the routine radionuclide data validation checklist, Part IIIb; and

6.4.3.3 go to Section 6.5, Verify Laboratory Control Sample Results.

6.4.4 If the tracer/carrier %R value *is not between* 10% and 30%, inclusive,

6.4.4.1 record “N” in block 2a of the routine radionuclide data validation checklist, Part IIIb;

6.4.4.2 record “n/a” in block 2c of the routine radionuclide data validation checklist, Part IIIb; and

6.4.4.3 go to Section 6.4.6.

6.4.5 If the tracer/carrier %R value *is between* 10% and 30%, inclusive,

6.4.5.1 record “Y” in block 2a of the routine radionuclide data validation checklist, Part IIIb.

6.4.5.2 For *detected* results,

1) circle “J, R1a” in block 2b of the routine radionuclide data validation checklist, Part IIIb;

2) record the qualifier flag and reason code combination “J, R1a” next to the nuclide result, on the Form I; and

3) record the affected samples in block 2c of the routine radionuclide data validation checklist, Part IIIb.

6.4.5.3 For *nondetected* results,

1) circle “UJ, R1b” in block 2b of the routine radionuclide data validation checklist, Part IIIb;
2) record the qualifier flag and reason code combination “UJ, R1b” next to the nuclide result, on Form I; and
3) record the affected samples in block 2c of the routine radionuclide data validation checklist, Part IIIb.

6.4.5.4 Record “N” in block 3a of the routine radionuclide data validation checklist, Part IIIb and

6.4.5.5 record “n/a” in block 3c of the routine radionuclide data validation checklist, Part IIIb.

6.4.6 If the tracer/carrier %R value is less than 10%,

6.4.6.1 record “Y” in block 3a of the routine radionuclide data validation checklist, Part IIIb;

6.4.6.2 circle “R, R1” in block 3b of the routine radionuclide data validation checklist, Part IIIb;

6.4.6.3 record the qualifier flag and reason code combination “R, R1” next to the nuclide result, on Form I; and

6.4.6.4 record the affected samples in block 3c of the routine radionuclide data validation checklist, Part IIIb.

6.4.6.5 record “N” in block 2a and “n/a” in block 2c of the routine radionuclide data validation checklist, Part IIIb.

6.4.7 Use the logic diagram of Figure 6.4-1 to determine which, if any, LANL qualifier flags and reason codes the **validator** must assign to the sample results for noncompliant tracer/carrier recoveries.

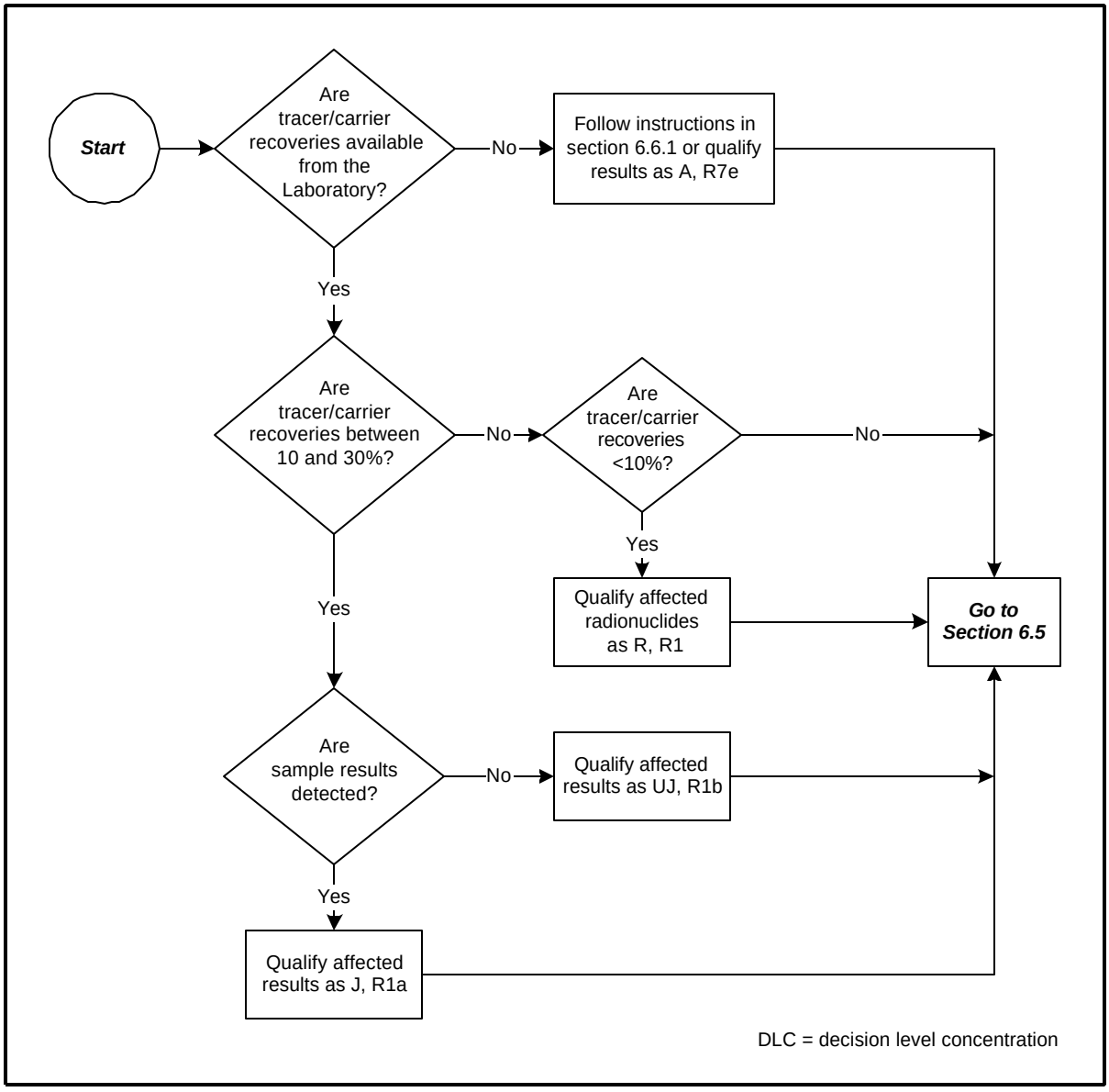

Figure 6.4-1. Assigning LANL qualifier flags and reason codes to the sample results for noncompliant tracer/carrier recoveries.

6.5 Verify Laboratory Control Sample Results

Note: Section 6.5 applies to all routine radionuclide analysis methods and analytes. Verify the presence of the LCS %R values using forms provided by the analytical laboratory.

6.5.1 If a method-specific LCS *was analyzed* with this RN,

6.5.1.1 record "N" in block 1a, of the routine radionuclide data validation checklist, Part IV;

6.5.1.2 record "n/a" in block 1c of the routine radionuclide data validation checklist, Part IV; and

6.5.1.3 go to Section 6.5.3

6.5.2 If a method-specific LCS *was not analyzed* with this RN,

6.5.2.1 record “Y” in block 1a, of the routine radionuclide data validation checklist, Part IV;

6.5.2.2 circle "A, R6e" in block 1b of the routine radionuclide data validation checklist, Part IV;

6.5.2.3 record the qualifier flag and reason code combination “A, R6e” next to the result for each affected target analyte, on Form I; and

6.5.2.4 record the LCS or LCS analytes not analyzed with this RN in block 1c of the routine radionuclide data validation checklist, Part IV.

6.5.3 If the LCS %R values for *all* analytes fall between 80% and 120%, inclusive,

6.5.3.1 record “N” in blocks 2a and 3a of the routine radionuclide data validation checklist, Part IV;

6.5.3.2 Record “n/a” in blocks 2c, 2d, 3c and 3d of the routine radionuclide data validation checklist, Part IV; and

6.5.3.3 go to Step 6.6, Verify Matrix-Spike Results.

6.5.4 If *no* LCS analyte recovery is less than 80% then:

6.5.4.1 record “N” in block 2a of the routine radionuclide data validation checklist, Part IV;

6.5.4.2 record “n/a” in blocks 2c and 2d of the routine radionuclide data validation checklist, Part IV; and

6.5.4.3 go to Section 6.5.6.

6.5.5 If *any* LCS analyte %R value is less than 80%,

6.5.5.1 record “Y” in block 2a of the routine radionuclide data validation checklist, Part IV;

6.5.5.2 circle, in block 2b of the routine radionuclide data validation checklist, Part IV,

1) “J-, R6a” for detected sample analytes and

2) “UJ, R6b” for non-detected analytes;

6.5.5.3 record, next to the result for each affected target analyte, on Form I, the qualifier flag and reason code combination

1) “J-,R6a” for detected and

2) "UJ,R6b" for non-detected analytes;

6.5.5.4 record the noncompliant LCS analytes in block 2c of routine radionuclide data validation checklist, Part IV; and

6.5.5.5 record the %R values of the noncompliant LCS analytes in block 2d of routine radionuclide data validation checklist, Part IV.

6.5.6 If *no* LCS analyte %R value is greater than 120%,

6.5.6.1 record "N" in block 3a of the routine radionuclide data validation checklist, Part VI;

6.5.6.2 record "n/a" in blocks 3c and 3d of the routine radionuclide data validation checklist, Part VI; and

6.5.6.3 go to Section 6.6, Verify Matrix-Spike Results.

6.5.7 If *any* LCS analyte %R value is greater than120%,

6.5.7.1 record "Y" in block 3a of the routine radionuclide data validation checklist, Part VI;

6.5.7.2 circle, in block 3b of the routine radionuclide data validation checklist, Part VI,

1) "J+, R6" for *detected* sample analytes and

2) no qualifier for *nondetected* sample analytes;

6.5.7.3 record, next to the result for each affected target analyte, on Form I, the qualifier flag and reason code combination

1) "J+, R6" for detected analytes and

2) no qualifier for *nondetected* sample analytes;

6.5.7.4 record the noncompliant LCS analytes in block 3c of routine radionuclide data validation checklist, Part VI; and

6.5.7.5 record the %R values of the noncompliant LCS analytes in block 3d of routine radionuclide data validation checklist, Part VI.

6.5.8 Use the logic diagram of Figure 6.5-1 to determine which, if any, LANL qualifier flags and reason codes the **validator** must assign to the sample results for noncompliant LCS analytes.

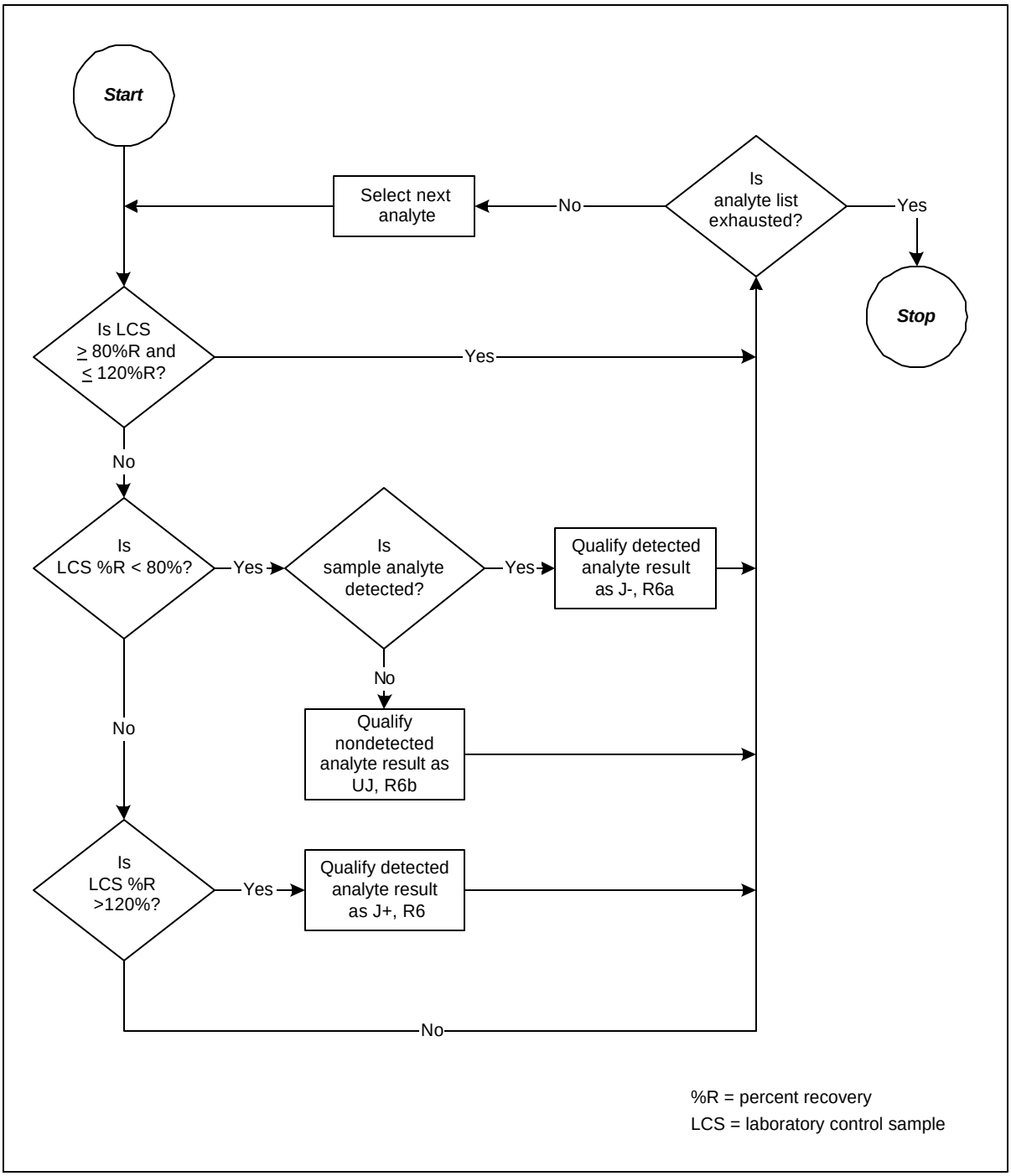

Figure 6.5-1. Assigning LANL qualifier flags and reason codes to the sample results for noncompliant LCS analytes.

6.6 Verify Matrix-Spike Results

Note: Section 6.6 applies only to GPC analysis for strontium-90. For all other routine radionuclide methods, go to Section 6.7. For strontium-90, verify the presence of the matrix-spike sample recoveries using the forms (CLP form V equivalent) provided by the analytical laboratory. The matrix-spike

acceptance criteria are 75%–125%, inclusive, for all spiked analytes If the sample result is greater than four times the spike added, these acceptance criteria do not apply.

6.6.1 If a matrix spike *was analyzed* on a sample associated with this RN,

6.6.1.1 record “Y” in block 1a of routine radionuclide data validation checklist, Part Va;

6.6.1.2 record “n/a” in block 1c of routine radionuclide data validation checklist, Part Va; and

6.6.1.3 go to Section 6.6.3.

6.6.2 If a matrix spike *was analyzed* on a sample *not associated* with this RN *and* no matrix spike was analyzed on a sample associated with this RN,

6.6.2.1 record “N” in block 1a of the routine radionuclide data validation checklist, Part Va;

6.6.2.2 circle "PM, R14b” in block 1b of the routine radionuclide data validation checklist, Part Va;

6.6.2.3 record the qualifier flag and reason code combination "PM, R14b” next to each affected strontium-90 result, on Form I;

6.6.2.4 if the matrix-spike sample *was* an ER Project sample, record the RN and sample ID of the spike sample in block 1c of the routine radionuclide data validation checklist, Part Va; and

6.6.2.5 if the matrix spike sample *was not* an ER Project sample, note this in block 1c and the %R value in block 1d of the routine radionuclide data validation checklist, Part Va.

6.6.3 If insufficient sample volume was submitted for analysis and *no* matrix spike could be analyzed,

6.6.3.1 record “N” in block 1a of the routine radionuclide data validation checklist, Part Va;

6.6.3.2 circle “A, R14a” in block 1b of the routine radionuclide data validation checklist, Part Va; and

6.6.3.3 record the qualifier flag and reason code combination “A, R14a” next to each affected strontium-90 result, on Form I.

6.6.4 If *all* matrix-spike %R values meet the acceptance criteria (75%–125%) *or* the sample result is greater than four times the spike amount,

6.6.4.1 record “N” in blocks 2a and 3a of the routine radionuclide data validation checklist, Part Vb;

6.6.4.2 record “n/a” in blocks 2c, 2d, 3c, and 3d of the routine radionuclide data validation checklist, Part Vb; and

6.6.4.3 go to Step 6.7, Assemble the Validation Data Record Package.

6.6.5 If *any* matrix-spike %R value is greater than125%,

6.6.5.1 record “Y” in block 2a of the routine radionuclide data validation checklist, Part Vb.

6.6.5.2 If the target analyte *is detected* in the sample,

1) circle “J+, R3” in block 2b of the routine radionuclide data validation checklist, Part Vb;

2) record the qualifier flag and reason code combination “J+, R3” next to the sample results for detected strontium-90, on Form I;

3) record the affected samples in block 2c of the routine radionuclide data validation checklist, Part Vb; and

4) record the %R values of strontium-90 that do not meet the acceptance criterion in block 2d of the routine radionuclide data validation checklist, Part Vb.

6.6.5.3 If the target analyte *is not detected* in the sample,

1) record the affected samples in block 2c of the routine radionuclide data validation checklist, Part Vb and

2) record the %R values of strontium-90 that do not meet the acceptance criterion in block 2d of the routine radionuclide data validation checklist, Part Vb.

6.6.6 If *any* matrix-spike %R value is less than 75%,

6.6.6.1 record “Y” in block 3a of the routine radionuclide data validation checklist, Part Vb.

6.6.6.2 If the target analyte *is detected* in the sample,

1) circle “J-, R3a” in block 3b of the routine radionuclide data validation checklist, Part Vb and

2) record the qualifier flag and reason code combination “J- ,R3a” next to the sample result for each affected target analyte, on Form I.

6.6.6.3 If the target analyte *is not detected* in the sample,

1) circle “UJ, R3c” in block 3b of the routine radionuclide data validation checklist, Part Vb and

2) record the qualifier flag and reason code combination “UJ, R3c” next to each affected result, on Form I.

6.6.6.4 Record the affected samples in block 3c of the routine radionuclide data validation checklist, Part VII and

6.6.6.5 record the %R values of strontium-90 that do not meet the acceptance criterion in block 3d of the routine radionuclide data validation checklist, Part VII.

6.6.7 Use the logic diagram of Figure 6.6-1 to determine which, if any, LANL qualifier flags and reason codes the **validator** must assign to the sample results for noncompliant matrix-spike analytes.

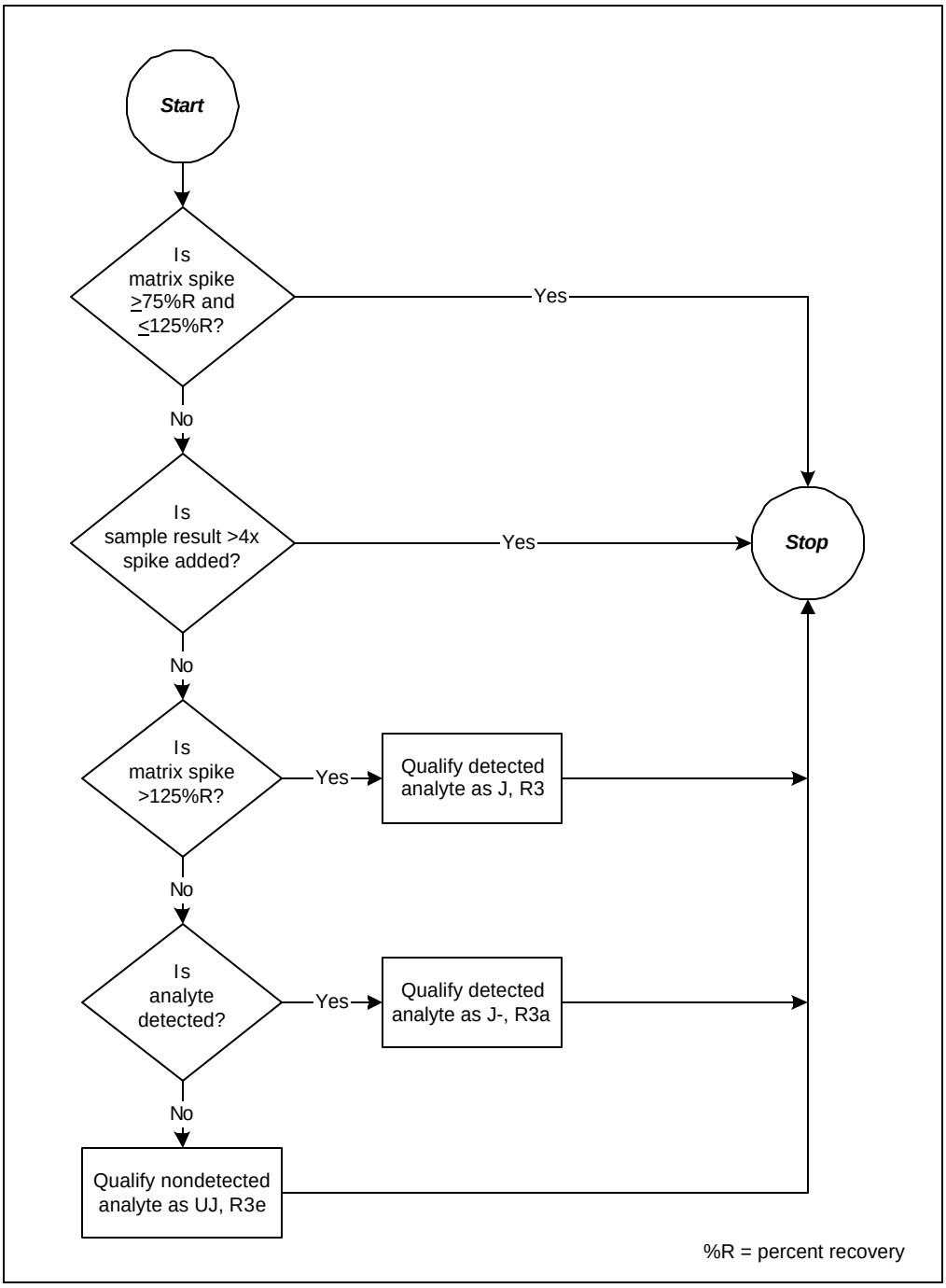

Figure 6.6-1. Assigning LANL qualifier flags and reason codes to the sample results for noncompliant matrix-spike analytes.

6.7 Assemble the validation data record package to include the following items in the order they are listed below:

6.7.1 the completed, signed, and dated data validation cover sheet;

6.7.2 the routine radionuclide data validation checklist forms completed in Sections 6.2 through 6.6;

6.7.3 photocopies of the completed forms (Form I's) on which the data validator recorded the qualifier flags and reason codes;

6.7.4 a photocopy of the data record package case narrative; and

6.7.5 photocopies of the data record package QC forms (assemble in order by QC forms).

6.8 Submit the validation data record package to the SMO, in accordance with ER-SOP-15.09.

7.0 REFERENCES

The following documents have been cited within this procedure:

ANSI, "Measurement and Associated Instrumentation Quality Assurance for Radioassay Laboratories," (Publisher, City, State, 1996). (ANSI N42.23-1996)

Currie, L., "Limits for Qualitative Detection and Quantitative Determination," *Analytical Chemistry*, (Vol. 40, No.3, pp. 586–593.) March, 1968

DOE (U.S. Department of Energy), December 1997. Multi-Agency Radiation Survey and Site Investigation Manual (MARSSIM), Final, Washington, D.C. (DOE 1997, 63128)

EPA (US Environmental Protection Agency), February 1994. "US EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review," Publication 9240.1-05-01, EPA-540/R-94/013, Office of Solid Waste and Emergency Response, Washington, DC.

ER-SOP-15.09, Chain of Custody for Analytical Data Packages

Fong, S., and J. Alvarez, "Data Quality Objectives for Surface-Soil Cleanup Operation Using *In Situ* Gamma Spectrometry for Concentration Measurements," *Health Physics*, (Vol. 72, No.2) February 1997.

LANL (Los Alamos National Laboratory), 1995. Environmental Restoration Statement of Work for Analytical Services, Los Alamos National Laboratory October, 1995.

QP-2.2, Personnel Orientation and Training

QP-4.2, Standard Operating Procedure Development

8.0 RECORDS

Although no records will be submitted to the Records Processing Facility (RPF) in the course of completing this procedure, the items identified in Section 6.8 will be a part of the data record package submitted to the RPF from the SMO in accordance with ER-SOP-15.09.

9.0 ATTACHMENTS

The document user may employ documentation formats different from those attached to/named in this procedure—as long as the substituted formats in use provide, as a minimum, the information required in the official forms developed by the procedure.

Attachment A: Laboratory Data Validation Qualifier Flags (1 page)

Attachment B: Chemical Separation Alpha Spectrometry, Gas Proportional Counting, and Liquid Scintillation Data Validation Reason Codes (2 pages)

Attachment C: Data Validation Cover Sheet (1 page)

Attachment D: Chemical Separation Alpha Spectrometry, Gas Proportional Counting, and Liquid Scintillation Data Validation Checklist Part I – Part V (5 pages)

Laboratory Data Validation Qualifier Flags

A	The contractually required supporting documentation for this datum is absent.
U	The analyte is classified as “not detected.”
J	The analyte is classified as “detected” but the reported concentration value is expected to be more uncertain than usual.
J+	The analyte is classified as “detected” but the reported concentration value is expected to be more uncertain than usual with a potential positive bias.
J-	The analyte is classified as “detected” but the reported concentration value is expected to be more uncertain than usual with a potential negative bias.
UJ	The analyte is classified as “not detected” with an expectation that the reported result is more uncertain than usual.
RPM	The reported sample result is classified as “rejected” due to serious non-compliances regarding quality control acceptance criteria. The presence or absence of the analyte cannot be verified based on routine validation alone.
PM	Manual review of raw data is recommended to determine if the observed non-compliance(s) with quality acceptance criteria adversely impacts data use.

Note: A “PM” qualifier flag indicates that a manual review should be conducted if the datum that is qualified with the “PM” is important to the data user. In addition, "PM" also means that a decision must be made by the project manager/delegee regarding the need for further review of the data. This review should include some consideration of potential impact that could result from using the "PM" qualified data.

Chemical Separation Alpha Spectrometry, Gas Proportional Counting, and Liquid Scintillation Data Validation Reason Codes

R1	The tracer/carrier %R value is less than 10%.
R1a	The tracer %R value is 10–30% inclusive and the sample result is greater than the MDC.
R1b	The tracer %R value is 10–30% inclusive and the sample result is less than the MDC.
R1e	The tracer/carrier %R value is not reported.
R3	The matrix-spike %R value is greater than the upper limit and the sample result is greater than the MDC.
R3a	the matrix-spike %R value is less than the lower limit and the sample result is greater than the MDC.
R3c	The matrix-spike %R value is less than the lower limit and the sample result is less than the MDC.
R4	The sample result is greater than MDC but is less than five times the amount found in the blank.
R4b	Blank data is either missing from or not reported in the data record package.
R5	Analyte is not detected because the amount reported is less than the MDC.
R6	Recovery of analyte in the LCS is greater than the upper limit and the analyte is greater than the MDC in the sample.
R6a	Recovery of analyte in the LCS is less than the lower limit and the analyte is greater than the MDC in the sample.
R6e	LCS data is missing from data record package.
R7b	The duplicate and sample results have a DER (duplicate error ratio) that is greater than 2.0.
R14a	Insufficient sample volume was received for a matrix-spike analysis.
R14b	The matrix-spike analysis was not performed on a sample associated with this RN.

Data Validation Cover Sheet

Section I.

Request Number:_____ Validation Date:_____ Lab Code:_____

Contract Laboratory Name:_____

Validator:_____ Organization:_____

Analytical Suite (check all that apply): ☐ Volatile Organics ☐ Semivolatile Organics ☐ Organochlorine Pesticides/Polychlorinated Biphenyls ☐ High Explosives ☐ Inorganics ☐ Radiochemistry

Other (describe): _____

Section II. Completeness Check

Yes	No	n/a	(check one)	Yes	No	n/a	(check one)
☐	☐	☐	1. Chain-of-custody form(s)	☐	☐	☐	6. Raw/BSS data
☐	☐	☐	2. Case narrative	☐	☐	☐	7. Quality control forms
☐	☐	☐	3. Sample result forms	☐	☐	☐	8. Quantitation reports
☐	☐	☐	4. Sample chromatograms	☐	☐	☐	9. TICs forms
☐	☐	☐	5. Standard chromatograms	☐	☐	☐	10. TICs mass spectra

Identify any samples in the assigned Request Number that are missing:

_____ _____ _____ _____ _____ _____ _____ _____

_____ _____ _____ _____ _____ _____ _____ _____

Comments/problems noted (include information about requests for further information submitted to the contract laboratory and agreed upon date of resolution and contract laboratory point of contact):

(Attach additional comment sheets as necessary)

Validator's signature: _____ Date: _____

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Chemical Separation Alpha Spectrometry, Gas Proportional Counting, and Liquid Scintillation Data Validation Checklist

Part I. Sample Statistics Check Sheet

Criterion	Criterion true? (yes, no, or n/a)	Action if "criterion true?" = yes	Comment
Is the batch's MDC stated?	1a.	1b. Record the MDC in block 1c and go on to Section 6.2.5.	1c. MDC =
Is the batch's MDC missing?	2a.	2b. Estimate the MDC using the metric: MDC = 3 × TPU Record the estimated MDC in block 2c.	2c. Estimated MDC =
Is the sample value < the MDC?	3a.	3b. Assign the LANL qualifier and reason code combination "**U, R5**" to all affeted sample anlytes. Record all affected sample analytes in block 3c.	3c.
Is the DER for the nuclide and the duplicate values > 2?	4a.	4b. Assign the LANL qualifier and reason code combination "**R, R7b**" to all affected nuclide values.	4c.

Chemical Separation Alpha Spectrometry, Gas Proportional Counting, and Liquid Scintillation Data Validation Checklist

Part IIa. Method Blank Validation Criteria

Criterion	Criterion true? (yes or no)	Action if "criterion true?" = no Assign qualifier & reason code…	List affected matricies or batches.
Was a method blank analyzed for each sample matrix and batch?	1a.	1b. "**A, R4b**" for any missing documentation. In block 1c, record all sample matrices and/or analytical batches that did not include a method blank.	1c.

Part IIb. Method Blank Validation Criteria (continued)

Criteria	Criterion true? (yes or no)	Action if "criterion true?" = yes Assign qualifier & reason code…	List detected blank analyte(s) and affected samples.	Analyte concentration (pCi/g)
Is a target analyte detected in the method blank **AND** is the same target analyte detected in the sample result < five times the amount detected in the method blank?	2a.	2b. "**U, R4**" to affected sample analyte(s).	2c.	2d.

Chemical Separation Alpha Spectrometry, Gas Proportional Counting, and Liquid Scintillation Data Validation Checklist

Part IIIa. Tracer/Carrier Recovery Validation Criteria

Criterion	Criterion true? (yes or no)	Action if "criterion true?" = no Assign qualifier & reason code…	Comments
Is a tracer/carrier recovery result reported?	1a.	1b. "**A, R1e**" to all results associated with the missing tracer/carrier results.	1c.

Part IIIb. Tracer/Carrier Recovery Validation Criteria (continued)

Criterion	Criterion true? (yes or no)	Action if "criterion true?" = yes Assign qualifier & reason code…	Comments
Is the tracer/carrier recovery result between 10%–30%, inclusive?	2a.	2b. "**J, R1a**" to any *detected* nuclide results and "**UJ, R1b**" to any *nondetected* nuclide results.	2c.
Is the tracer/carrier recovery result less than 10%?	3a.	3b. "**R, R1**" to all results associated with the tracer/carrier recovery < 10%.	3c.

Chemical Separation Alpha Spectrometry, Gas Proportional Counting, and Liquid Scintillation Data Validation Checklist

Part IV. Laboratory Control Sample (LCS) Validation Criteria

Criteria	Criterion true? (yes/no)	Action if "criterion true?" = yes Assign qualifier & reason code…	List all noncompliant LCS analytes.	LCS %Rs
Was a required LCS *not* associated with this request?	1a.	1b. "**A, R6e**" for any missing documentation. In block 1c, record • the LCS or LCS analytes not reported.	1c.	1d. n/a
Is the LCS percent recovery value < 80% for any analyte?	2a.	2b. "**J-, R6a**" to all *detected* sample analytes and "**UJ, R6b**" to all *nondetected* sample analytes. In block 2c record • noncompliant LCS analytes. In block 2d record • the %R values of the noncompliant LCS analytes.	2c.	2d.
Is the LCS percent recovery value > 120% for any analyte?	3a.	3b. "**J+, R6**" to all *detected* sample analytes and do not qualify *nondetected* sample analytes. In block 3c record • noncompliant LCS analytes. In block 3d record • the %R values of the noncompliant LCS analytes.	3c.	3d.

Chemical Separation Alpha Spectrometry, Gas Proportional Counting, and Liquid Scintillation Data Validation Checklist

Part Va. Matrix-Spike Validation Criteria

Criteria	Criterion true? (yes/no)	Action if "criterion true?" = no Identify noncompliant matrix-spike recoveries ***and*** assign qualifier & reason code…	List all affected matrix-spike analytes and samples.	Percent recovery
Is the matrix-spike sample present?	1a.	1b. "**PM, R14b**" if the matrix-spike sample that was analyzed was on a sample not associated with this request number. In block 1c record • the request number and sample ID of spiked sample. **OR** "**A, R14a**" if the matrix-spike sample was missing because insufficient sample volume was received by the analytical laboratory for the duplicate analysis.	1c.	1d. n/a

Part Vb. Matrix-Spike Validation Criteria (continued)

Criteria	Criterion true? (yes/no)	Action if "criterion true?" = yes Identify noncompliant matrix-spike recoveries ***and*** assign qualifier & reason code…	List all affected matrix-spike analytes and samples.	Percent recovery
Is the matrix spike percent recovery value > 125%?	2a.	2b. "**J+, R3**" to all detected sample analytes.	2c.	2d.
Is the matrix spike percent recovery value < 75%?	3a.	3b. "**J-, R3a**" to all *detected* sample analytes and "**UJ, R3**" to all *nondetected* sample analytes.	3c.	3d.

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